

LA-UR-21-20421

Approved for public release; distribution is unlimited.

Title: Cold Testing for Characterization of Oxide Feed Impurities in Support of the ARIES Program

Author(s): Schake, Ann Rene
Chacon, Enriquez
Stritzinger, Jared Tyler

Intended for: Report

Issued: 2021-01-19

Disclaimer:

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by Triad National Security, LLC for the National Nuclear Security Administration of U.S. Department of Energy under contract 89233218CNA000001. By approving this article, the publisher recognizes that the U.S. Government retains nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes. Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy. Los Alamos National Laboratory strongly supports academic freedom and a researcher's right to publish; as an institution, however, the Laboratory does not endorse the viewpoint of a publication or guarantee its technical correctness.

Cold Testing for Characterization of Oxide Feed Impurities in Support of the ARIES Program

Ann Schake, Enriquez Chacon, Jared Stritzinger

Overview

The Advanced Recovery and Integrated Extraction System (ARIES) Program converts surplus plutonium metal components into an oxide powder form and packages the material for final disposition. In support of the ARIES program, over a metric ton (MT) of plutonium has been removed from surplus nuclear weapons since 1998 and packaged for long-term storage in 3013 cans. The oxide production rate is anticipated to increase substantially over the next decade in support of dilute and dispose goals. Analytical chemistry data from destructive analysis of the resulting 70+ blend lots to date has been reported for contaminants in the product oxide. Changes in the Pu product specification for Pu oxide product, induced by a different pit mix, the LANL swap program and processing Alternate (non-Pit Pu) Feed Stock (AFS) metals, will affect the level and the mix of these contaminants in the upcoming years. With these changes in feed material, it will be important to provide fast and efficient measurement of elemental impurities in the oxide to be certain that the process is under control and to monitor the anticipated changes from the historical blend lot data. Non-destructive methods need to be evaluated for the characterization of the plutonium oxide product and the quantification of the impurities at the current and anticipated levels. A feasibility study has begun to investigate the applicability of new spectroscopic methods for oxide impurity characterization. To this end, here we describe the preparation of surrogate materials to support these investigations, and the results of a feasibility study investigating the use of LIBS (Laser Induced Breakdown Spectroscopy) to characterize the surrogate materials as calibration standards.

Table 1 shows the analytical chemistry data for the top impurities collected for the current 70+ blend lots. Based on the changes in the future lots, the levels of the contaminants are also expected to change. For example, it is anticipated that Ga and Be contaminant levels will increase substantially. It is also anticipated that RCRA metals will be more of a concern and the anions Cl^- and F^- and their counter ions (e.g. Na^+ , K^+ , Mg^{2+} , Ca^{2+}).

Table 1. ARIES Oxide Impurities

Element	Maximum Concentration ($\mu\text{g/g Pu}$)	Average Concentration ($\mu\text{g/g Pu}$)
Ga	11000	7764
U	4940	1600
Fe	1500	469
Cr	476	150
Si	290	78.5
Am	5640	4357
Ni	390	45.7
Np	270	200
C	190	84.6
Be	74	57

F	64.6	64.6
Cl	140	35

Based on these expectations, cold standards were prepared in a Bi₂O₃ matrix to test the feasibility of employing real time spectroscopic methods for the analysis of the plutonium oxide products. Standards in the surrogate matrix were prepared according to the target concentrations shown in Table 2, which encompass the current and anticipated levels for these contaminants. The standards were prepared gravimetrically and verified when available, as described below. The standards were then analyzed using a Hand-Held LIBS instrument, calibration curves prepared, and LODs determined from the data. Data collected during the cold testing of surrogate materials and mixtures will provide the technical basis for the development of work authorizing documents (IWD, DOP) for performing this work with plutonium oxide products in PF4 work spaces in the future.

Table 2. Target Element Concentrations

Element	Low conc Target (µg/g Pu)	Mid conc Target (µg/g Pu)	Mid 1 conc Target (µg/g Pu)	Mid 2 conc Target (µg/g Pu)	High conc Target (µg/g Pu)
Ag	0.25	0.5	0.75	10	100
Al	15	30	45	60	100
As	1	2.5	5	10	100
Ba	10	50	100	500	1000
C	1	10	100	500	1000
Cd	0.1	0.5	1	10	100
Cl	10	25	100	250	500
Cr	5	50	100	500	1000
F	10	25	100	250	500
Fe	10	100	500	1000	2000
Ga	500	1000	10000	15000	20000
Hg	0.05	0.1	0.2	1	100
K	1	5	10	100	1000
Mg	1	5	10	50	100
Na	5	25	50	100	150
Ni	10	50	100	250	500
Pb	1	5	10	100	500
Si	50	100	250	300	500
Se	0.25	0.5	0	10	50

In some cases, mixtures of elements were used. For example, a BaF₂ standard was prepared and employed for calibration curves for Ba and F, NaCl standards were prepared and curves determined for Na and Cl, K₂CO₃ standards were prepared and curves determined for K and C. Graphite was used for carbon surrogate samples, and elemental Si was used for the Si standards. In all other cases, oxides of the analyte were prepared as the standards. In addition, due to analytical chemistry requirements for carbon analysis, a series of Carbon standards was also prepared in a CeO₂ matrix. The oxides and salts used for the analytes were purchased from Sigma-Aldrich and Alfa-Aesar at >99% purity.

Sample Preparation and LIBS Measurements

In many cases, the target concentrations for the analytes were very low. Therefore, to avoid having to prepare 100s of grams of several of the mixtures, the higher concentration standard was prepared and mixed, followed by preparing serial dilutions of the standard for the lower concentrations. A micro-analytical balance was used to weigh the analyte directly into the polystyrene mixing vial, then Bi_2O_3 was added to the vial, determining the Bi_2O_3 weight by difference. A polystyrene mixing ball was added to the vial, and the mixture mixed for up to 15 minutes using a ChemPlex SpectroMill Ball Pestle Impact Grinder/Mixer. Pellets were pressed for each of the mixtures (~1.2 – 1.3 g) at ~8 tons using a 13mm Stainless Steel die in a ChemPlex Series T25 Semi-Automatic 25 Ton Spectro Press. See Figures 1 and 2 below.



Figure 1. Equipment and samples from surrogate standard preparation L to R: Pellet press, standard sample pellets, standard mixtures, Ball Pestle Impact Grinder/Mixer.



Figure 2. Left: Example of prepared sample pellet. Right: Collection of standard pellets for analysis

Most of the resulting mixtures were sampled and shipped to C-CDE for destructive chemical analysis by ICP-MS and ICP-OES to determine a measured concentration value to use in the standard curves and the final calibration curve. Unfortunately, only a few of the analytes could be analyzed and measured by C-CDE due to solubility issues and separation issues with the Bi_2O_3 matrix. A report from C-CDE with detailed results was provided.¹ Chemistry results for ten of the sample sets were reported that included: As, Cd, Ni, Mg, Hg, Ga, K, Na, Fe, and Si. These concentrations were used for the standard curves and Limit of Detection (LOD) determinations, with the exception of the Hg, Ni and Si samples. Only one of Hg samples was above the detection limit in C-CDE's measurements, and therefore none of the values were used. The reported values

for Ni were all ~50% of the expected values, and therefore were not used to prepare the standard curve and the LOD calculations. C-CDE had trouble with the Si samples precipitating out of solution on the ICP-OES columns, and therefore those values were also not used for the standard curve and LOD determinations. The BaF₂, NaCl and Carbon samples were sent to C-AAC for destructive analysis to determine F, Cl and C, respectively. The F and Cl results are still pending. The C sample in Bi₂O₃ and also CeO₂ were measured using a combustion technique. Several blank samples were also provided to C-AAC for their measurements and data analysis.

A SciAps Z300 Hand Held LIBS analyzer was employed for the LIBS analysis (Figure 3). Laser Induced Breakdown Spectroscopy (LIBS), a type of atomic optical emission spectroscopy (OES). LIBS works on the basic understanding that elements emit specific colors of light, which are a unique signature for each element. The wavelength (or color) of specific lines in the spectrum reveals the element present, and the intensity of the light at that wavelength is related to the concentration of each element. The Z300 laser system consists of up to 5-6 mJ of pulsed laser power (50 Hz, 1064 nm) and comes with a spectrometer that covers a spectral range of 190–950 nm. An argon purge was employed directly to the sample surface at the location of the laser-induced plasma. The standard SciAps acquisition settings were used for each analysis. Twelve sequential spots (50 µm) are analyzed in a 4x3 rectangular grid with 20 µm steps. Ten cleaning shots are fired at each location, followed by three shots for data collection. These three spectra were analyzed and averaged with the Profile Builder software. The data from the twelve locations is then averaged by the software and represent one data point for the ablated area. This analysis was performed at a minimum of five different locations for each standard mixture. Between each sample the laser nose piece was removed and the laser aperture cleaned with a cotton swab.



Figure 3. Picture of the SciAps Hand-Held LIBS instrument; and a schematic illustrating the first three steps of LIBS sample analysis: 1) plasma generation, 2) light emission, 3) light collection.

Using the Profile Builder Software, a standard curve for each analyte at their corresponding wavelengths was built using the masses and concentrations determined in the preparation of the mixtures, or the data from destructive analysis. Each spectral line was determined relative to Bi. An example of the Profile Builder data output for Ga is shown in Figure 4 below. A complete LIBS_Cal file was prepared by combining the measurements for all the analytes, representing the

calibration curve. For the LOD determinations, the spectral data was exported to Excel and a standard curve determined for each analyte. An individual standard curve was prepared for Carbon in CeO_2 following the same approach, with the data relative to Ce.

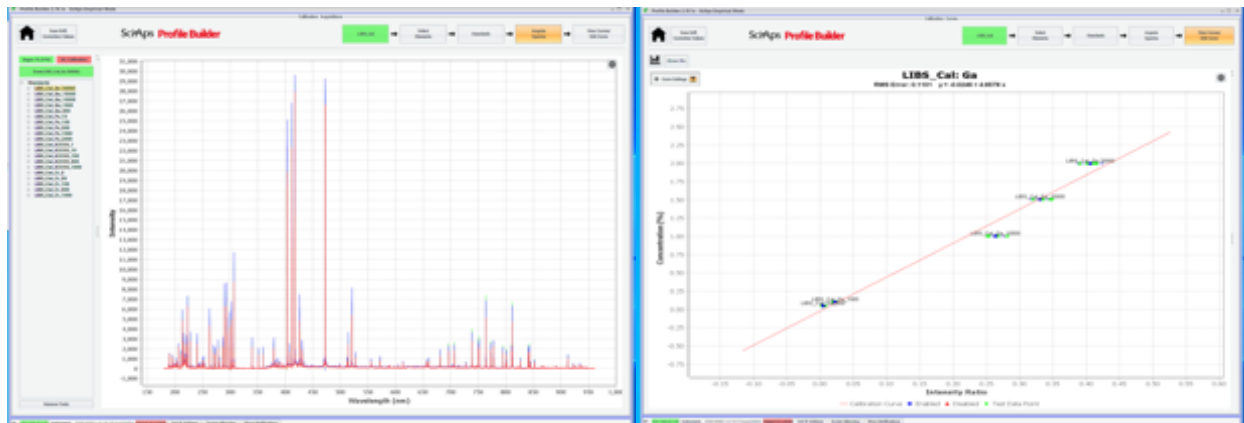


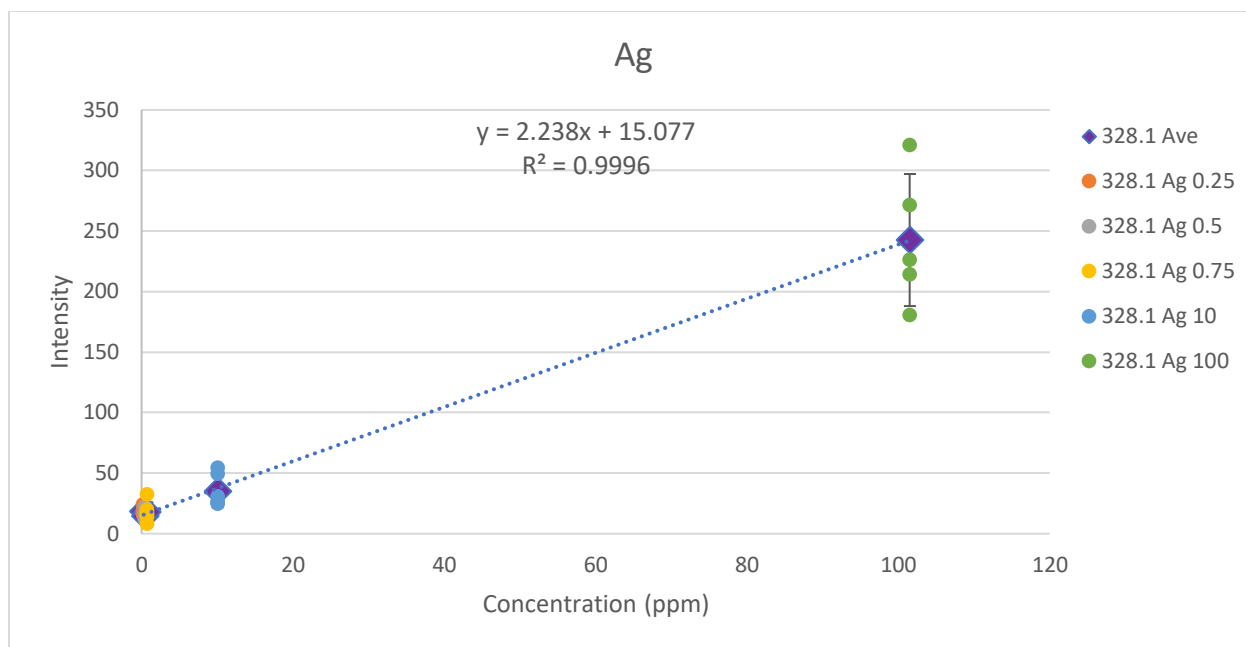
Figure 4. Ga spectra and Ga in Bi Standard Curve from Profile Builder software

Observations and Data Analysis

Observations for each element are summarized, along with recommendations for improving the data and the standard curve for each analyte if a better calibration curve and more reliable LODs are needed for the surrogate work. The standard curve for each element is also provided. The plots shown are either the intensity vs the analyte concentration or the intensity ratio of the analyte to Bi (or Ce for C) vs the analyte concentration.

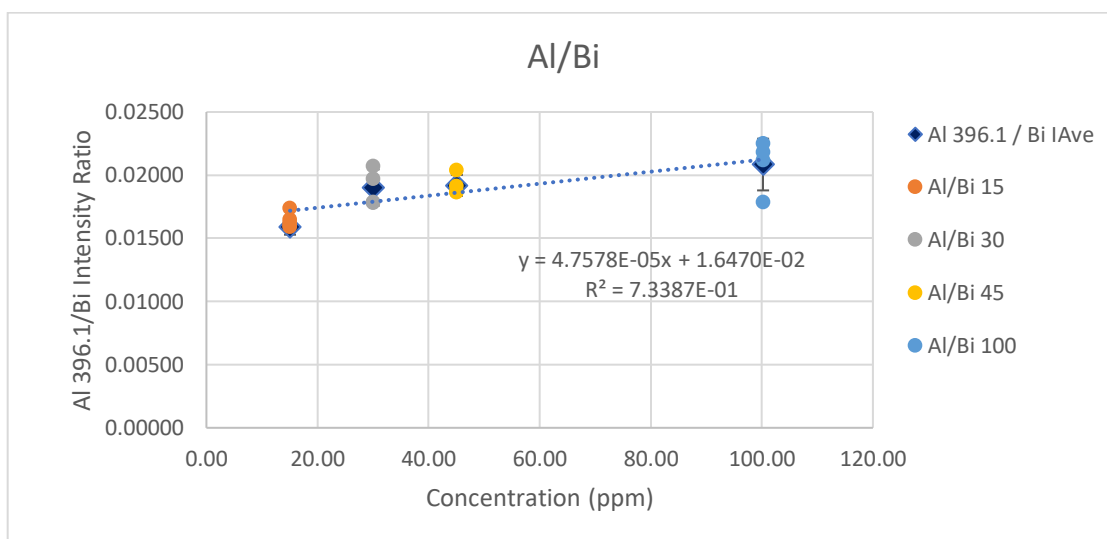
Silver, Ag

Silver oxide (AgO) mixtures were prepared in Bi_2O_3 at the target Ag/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the $\text{AgO}/\text{Bi}_2\text{O}_3$ mixtures. The standard curve shows a very good fit, however, using the masses and concentrations determined in the preparation of the mixtures. If improvements in LOD determinations are warranted, additional samples between 100 and 10 ppm and 10 and 1 ppm would be suggested.



Aluminum, Al

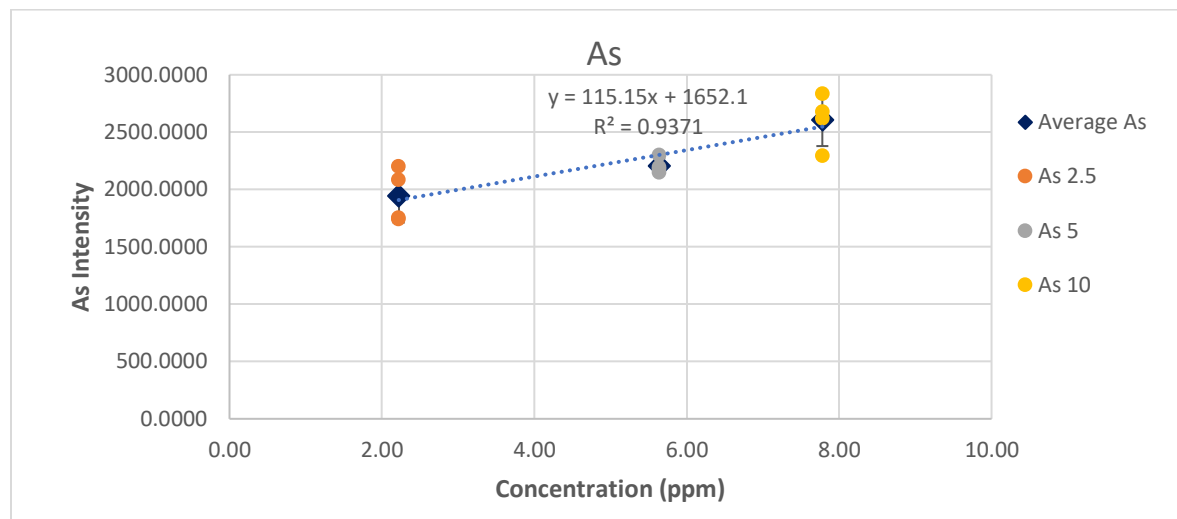
Aluminum oxide (Al_2O_3) mixtures were prepared in Bi_2O_3 at the target Al/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the standards. The standard curve for Al/Bi Intensity ratio is shown below, using the masses and concentrations determined in the preparation of the mixtures. The measurement for the intensity at 60 ppm was not included in the standard curve. Preparing and measuring a series of higher concentration standards to add to the curve would be beneficial in determining the LOD.



Arsenic, As

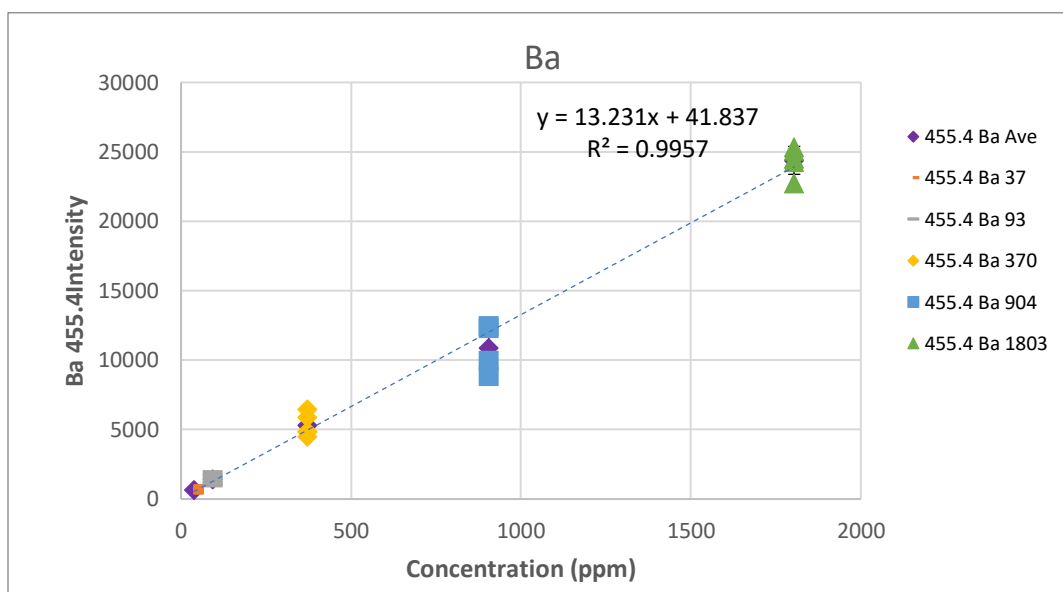
Arsenic oxide (As_2O_3) mixtures were prepared in Bi_2O_3 at the target As/Bi concentrations listed in Table 2. C-CDE was able to dissolve and analyze the As standards. Destructive analysis values

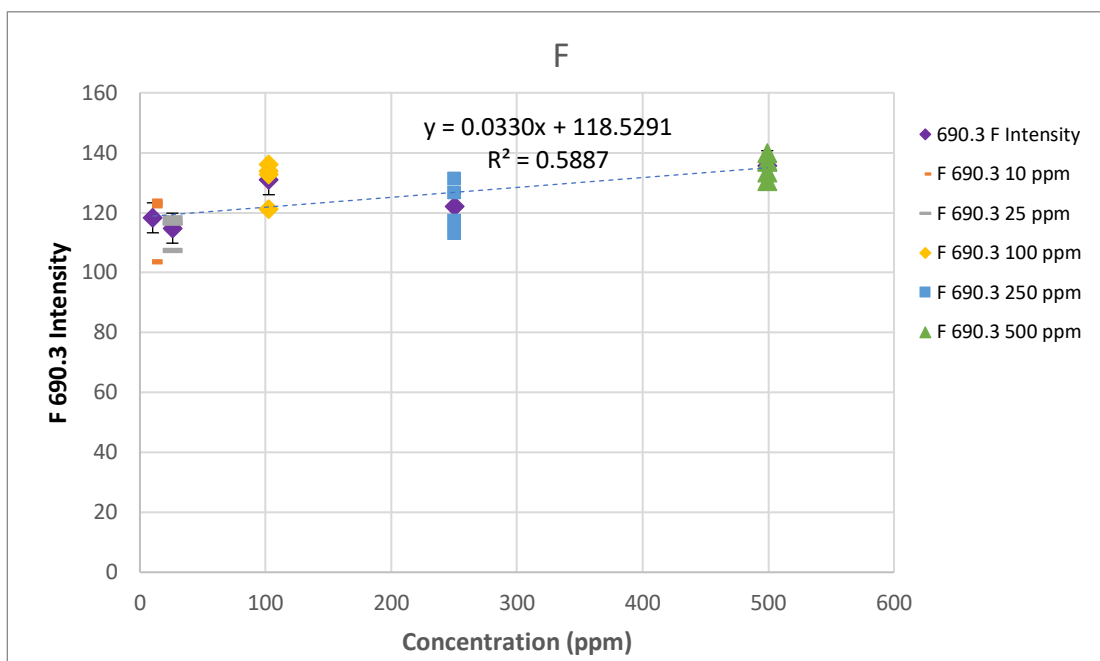
are reasonably close to the target values. The standard curve, however, was not very good unless the low and high concentration measurements were removed from the curve. Arsenic has two wavelengths to consider and they are both in areas that are interfered with by several other elements. To determine a better LOD, preparation and measurement of higher concentration standards to extend the standard curve might be useful. However, the results might not be improved due to the poor LIBS spectral lines.



Barium, Ba and Fluorine, F

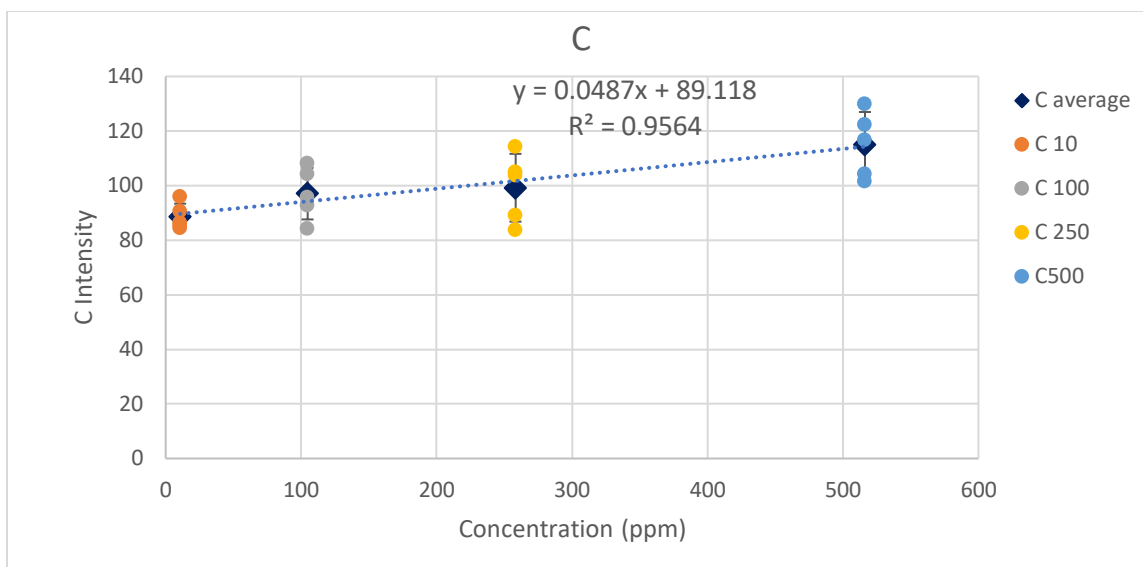
Barium Fluoride (BaF_2) mixtures were prepared in Bi_2O_3 at the target F/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the standards. The standard curve from the masses and concentrations determined in the preparation of the mixtures is shown below, and shows a very good fit. Samples of the BaF_2 mixtures were submitted to C-AAC for F analysis in May, but no data has been reported to date. Using the masses and concentrations determined in the preparation of the mixtures for F, the standard curve is very reasonable and shown below.





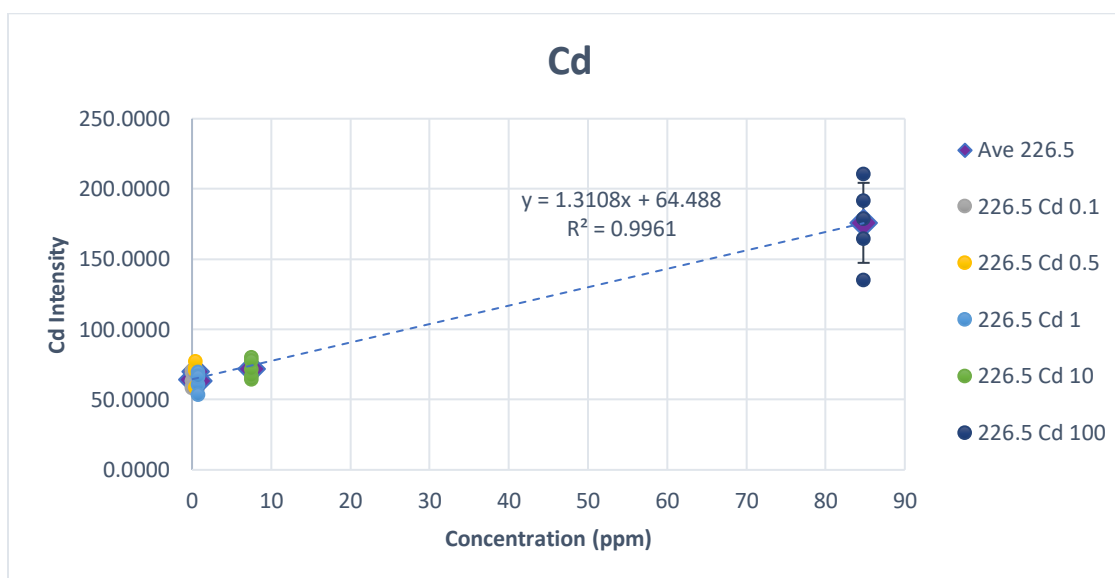
Carbon, C

Mixtures of K_2CO_3 in Bi_2O_3 were prepared using the target Carbon concentrations listed in Table 2. Samples of the mixtures were submitted to C-AAC for Carbon concentration determination by destructive analysis. Results from the analyses were not good due to Bi_2O_3 being a very good CO_2 getter.² Also, since the C method is a combustion technique, where C is converted to CO_2 , the K_2CO_3 salt was not a reasonable sample for this analysis. Samples of graphite were prepared in CeO_2 and submitted to C-AAC for a second series of analyses. The results of the carbon concentration analyses are very inconclusive. The CeO_2 showed a very high carbon blank, which was indicated in the sample results and also the blank results. The CeO_2 blanks were specifically prepared in the same way as the standards. Mixing the samples using the polystyrene vials and balls is believed to be adding to the C blank in the surrogate samples. However, the standard curve using the masses and concentrations determined in the preparation of the mixtures instead of the measured concentrations prepared in CeO_2 shows a reasonable fit and is shown below. (The 1000 ppm sample was not used in the standard curve.)



Cadmium, Cd

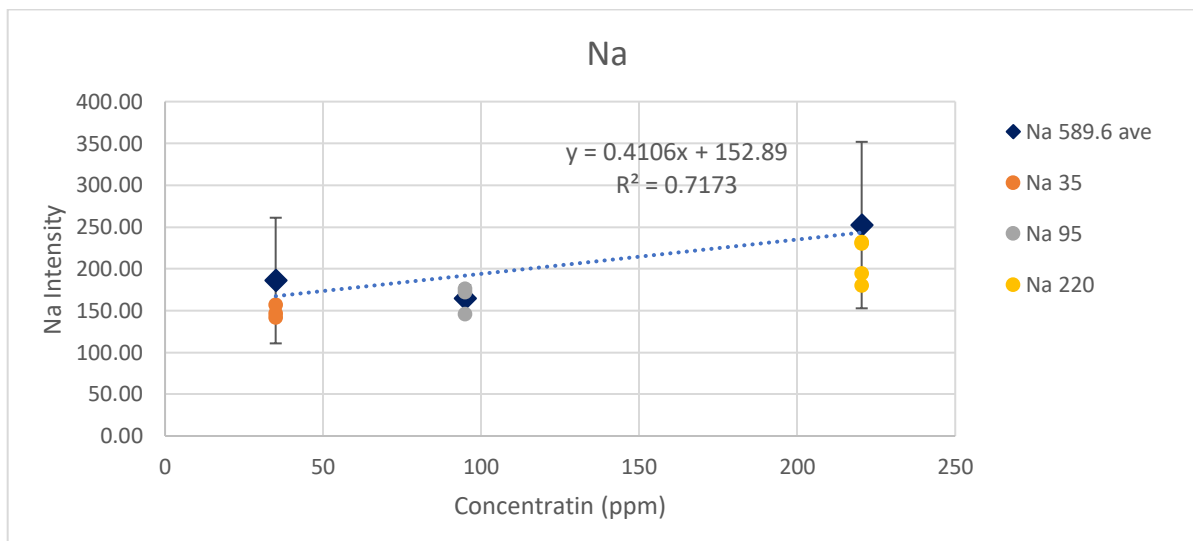
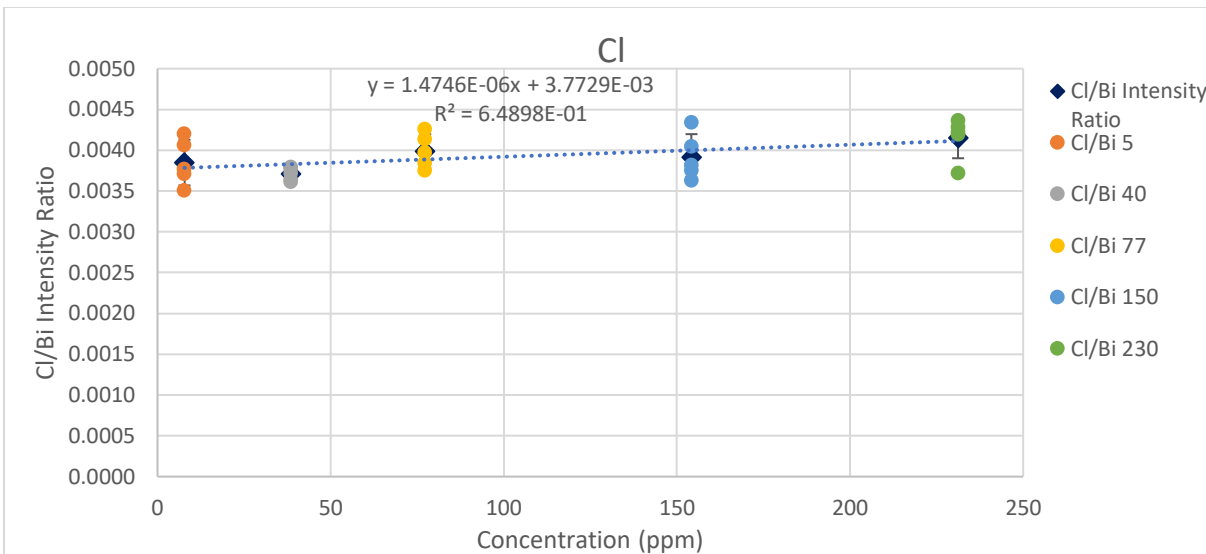
Cadmium oxide (CdO) mixtures were prepared in Bi_2O_3 at the target Cd/Bi concentrations listed in Table 2. C-CDE was able to dissolve and analyze the standards. Values obtained through destructive analysis are reasonably close to the target values and were used to prepare the standard curve. The standard curve shows a good fit and is shown below. Preparing and measuring a few points between 10 and 100 ppm to add to the curve, and extending the standard curve above 100 ppm would be beneficial in improving the LOD determination.



Chlorine, Cl and Sodium, Na

Sodium chloride (NaCl) mixtures were prepared in Bi_2O_3 at the target Na/Bi concentrations listed in Table 2. Samples of the NaCl/ Bi_2O_3 mixtures were submitted to C-AAC for Cl analysis in May but no data has been reported to date. It is important to note that Cl does not have any good wavelengths to consider in LIBS measurements. However, the calibration curve for Cl is

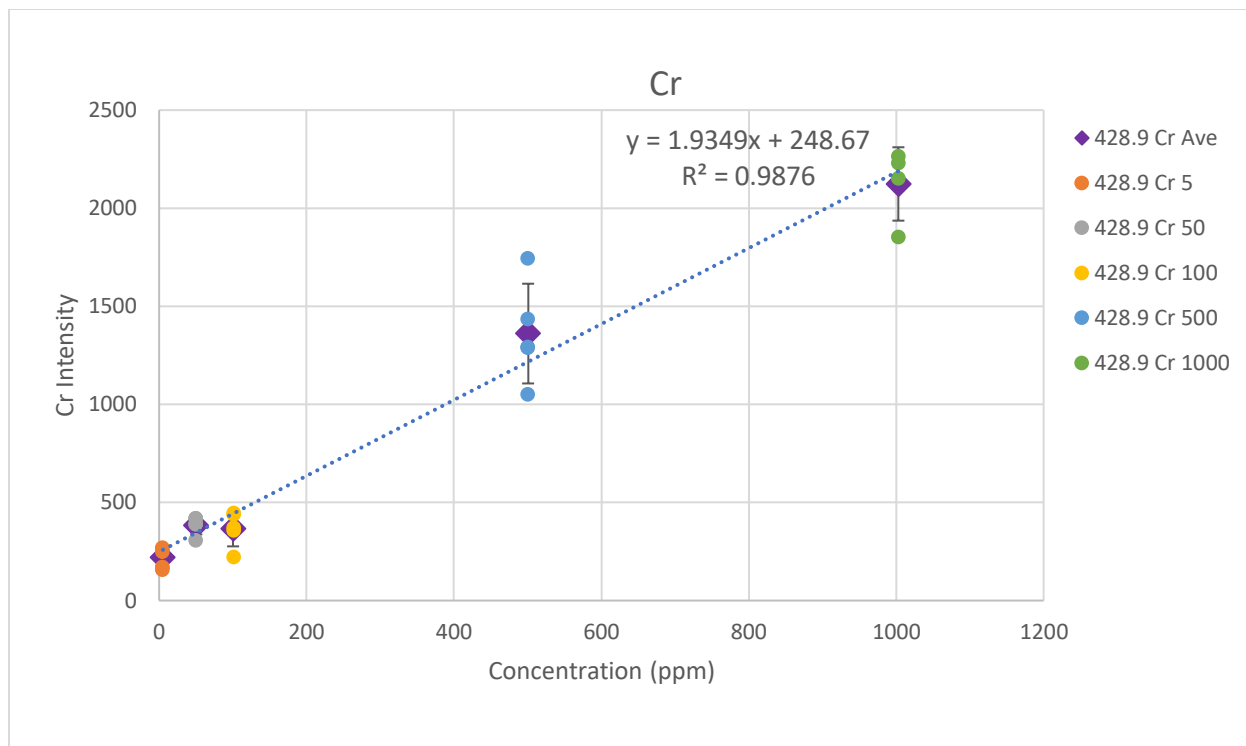
reasonably good using the masses and concentrations determined in the preparation of the NaCl mixtures and is shown below. The LOD determination for Cl could be improved by preparing higher concentrated samples, since the target values were not met due to using the Na target values in the preparation of the mixtures. C-CDE was able to dissolve and analyze the NaCl mixtures for Na. The values determined from destructive analysis are reasonably close to the target values and were used to prepare the standard curve. The standard curve for Na is good above 35 ppm. Values below 35 ppm were not used for the standard curve. Adding to the curve with higher concentration samples, and also preparing and measuring additional mixtures for the 50, 25 and 5 ppm samples could improve the LOD determinations.



Chromium, Cr

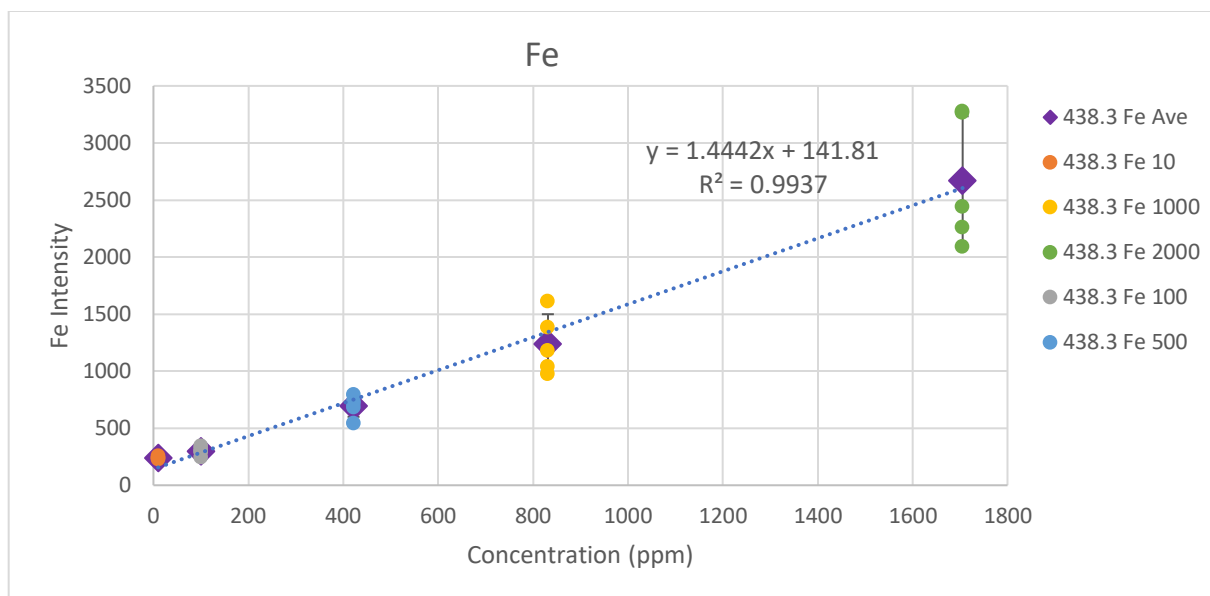
Chromium oxide (Cr_2O_3) mixtures were prepared in Bi_2O_3 at the target Cr/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the $\text{Cr}_2\text{O}_3/\text{Bi}_2\text{O}_3$ mixtures. The standard curve was prepared using the masses and concentrations determined in the preparation

of the mixtures. The curve is very good down to 50 ppm using these values. Preparing dilutions to provide measurements between 5 and 50 ppm to add to the standard curve could improve the LOD determinations.



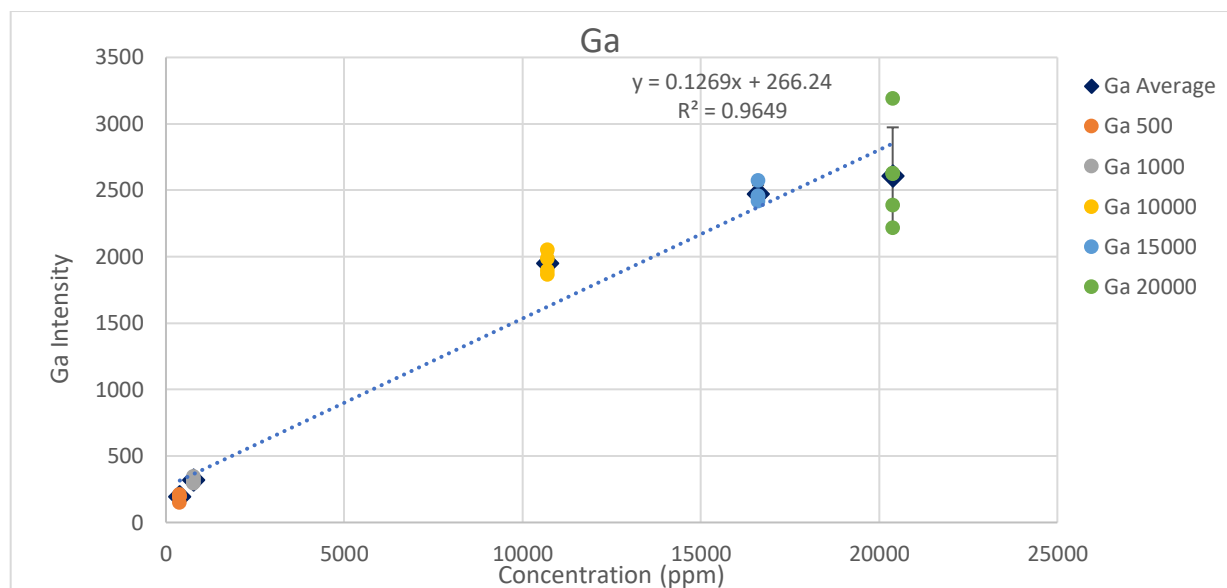
Iron, Fe

Ferric oxide (Fe_2O_3) mixtures were prepared in Bi_2O_3 at the target Fe/Bi concentrations listed in Table 2. C-CDE was able to dissolve and analyze the iron oxide standards. The results from destructive analysis are close to the target values and were used for the standard curve. The curve is very good and shown below.



Gallium, Ga

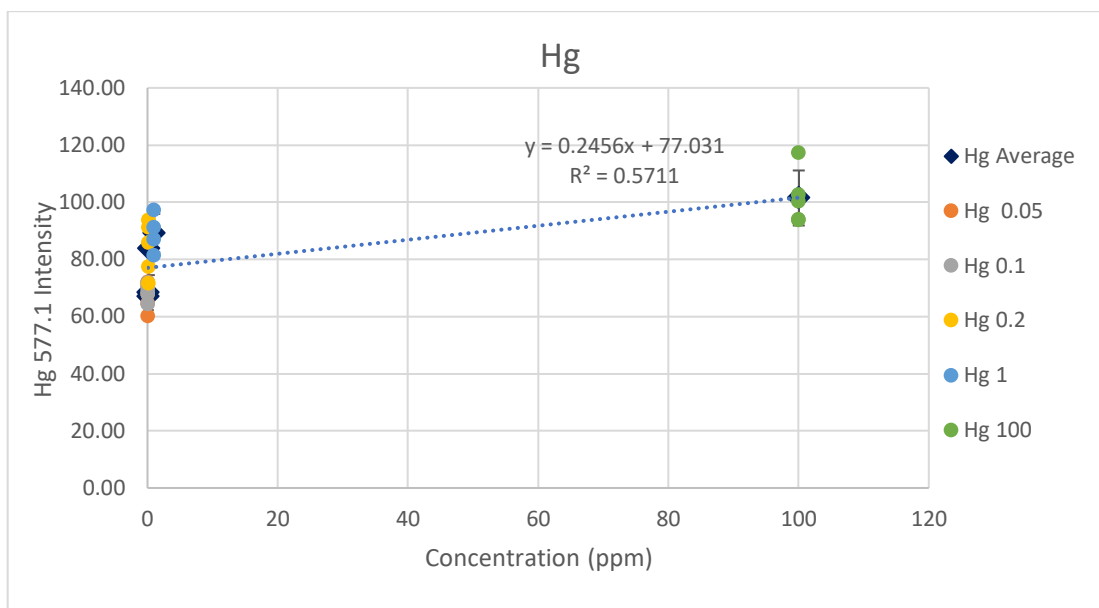
Gallium oxide (Ga_2O_3) mixtures were prepared in Bi_2O_3 at the target Ga/Bi concentrations listed in Table 2. C-CDE was able to dissolve and analyze the gallium oxide standards. The results from destructive analysis are close to the target values and were used for the standard curve. The curve shows a very good fit and is shown below. Lower concentration mixtures could be prepared and measured if needed to determine a better LOD.



Mercury, Hg

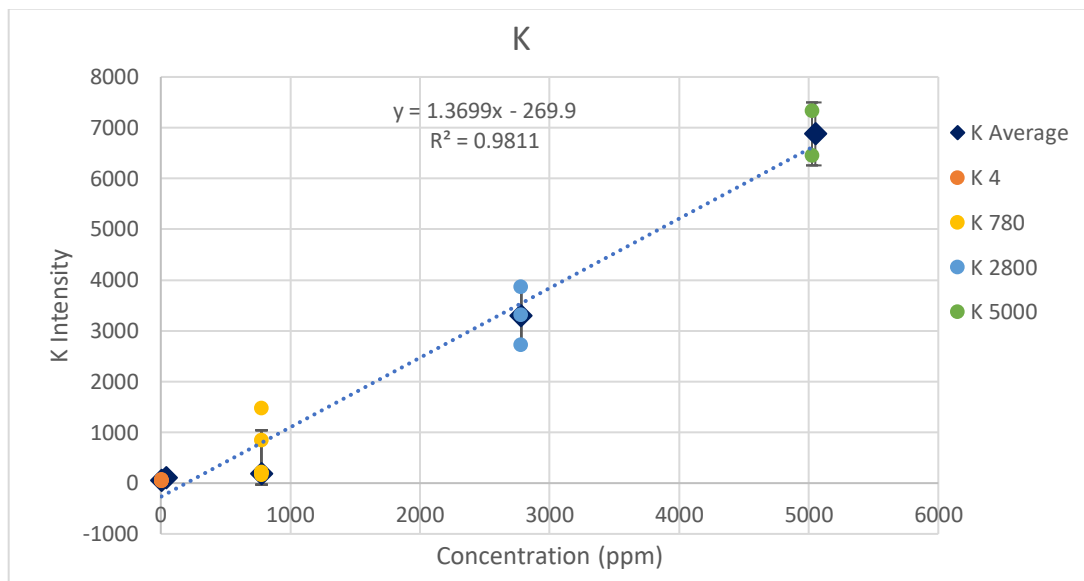
Mercuric oxide (HgO) mixtures were prepared in Bi_2O_3 at the target Hg/Bi concentrations listed in Table 2. C-CDE was able to dissolve the standards, however they were only able to measure the 100 ppm standard due to their MDL. The standard curve was prepared using the masses and concentrations determined in the preparation of the mixtures and is shown below. The fit to the

curve is not good due to the limited number of points. Preparing some dilutions to provide a few points between 10 and 100 ppm to add to the curve, and extending the standard curve above 100 ppm would be beneficial in improving the LOD determination.



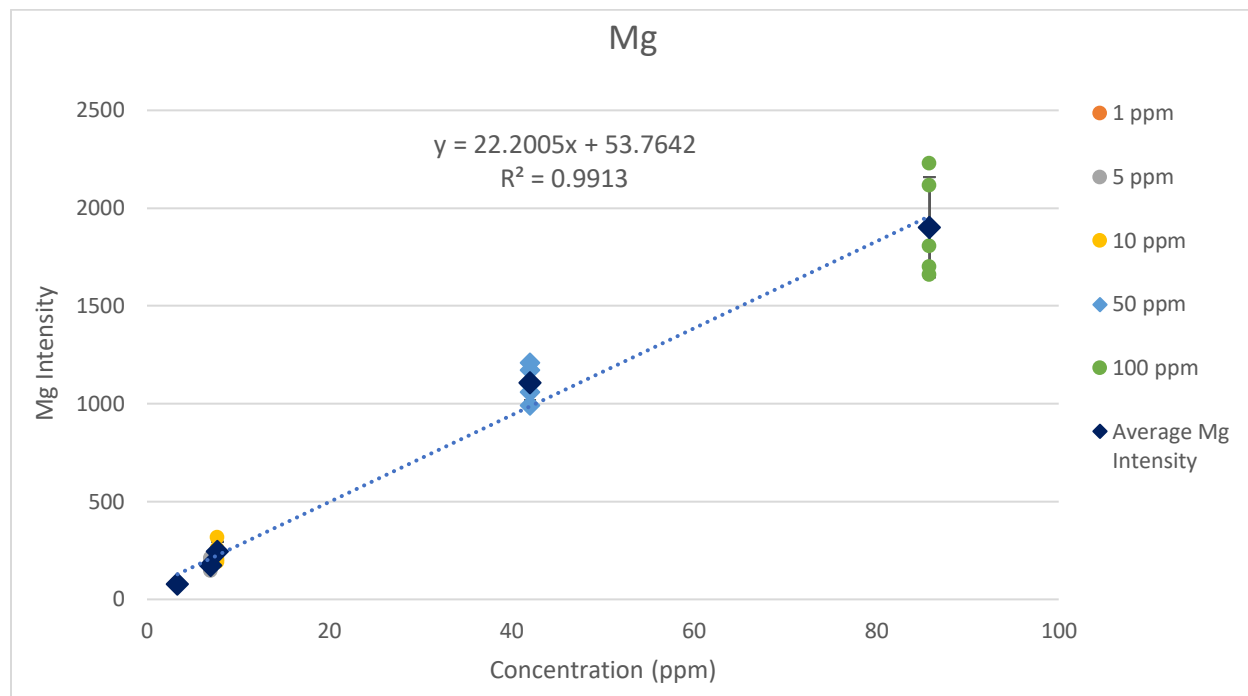
Potassium, K

Mixtures of K_2CO_3 in Bi_2O_3 were prepared using the target Carbon concentrations listed in Table 2. C-CDE was able to dissolve and analyze the standards. The results from destructive analysis are close to the target values and were used for the standard curve, shown below. Preparing dilutions to provide a few points between 100 and 1000 ppm to add to the current calibration line would be beneficial in improving the LOD determination. New mixtures of other potassium salts, e.g. KCl, could also be prepared and the K standard curve and LOD determinations done again.



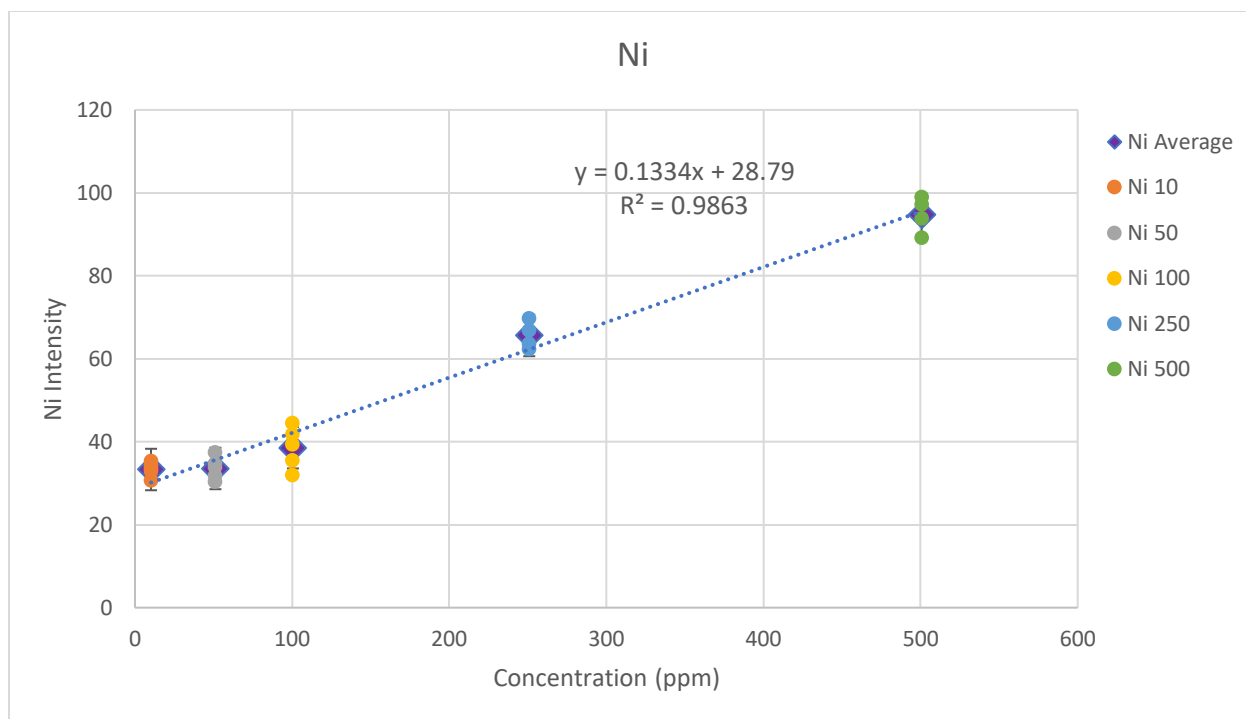
Magnesium, Mg

Magnesium oxide (MgO) mixtures were prepared in Bi₂O₃ at the target Mg/Bi concentrations listed in Table 2. C-CDE was able to dissolve and analyze the MgO standards. The results from destructive analysis are close to the target values and were used for the standard curve, shown below.



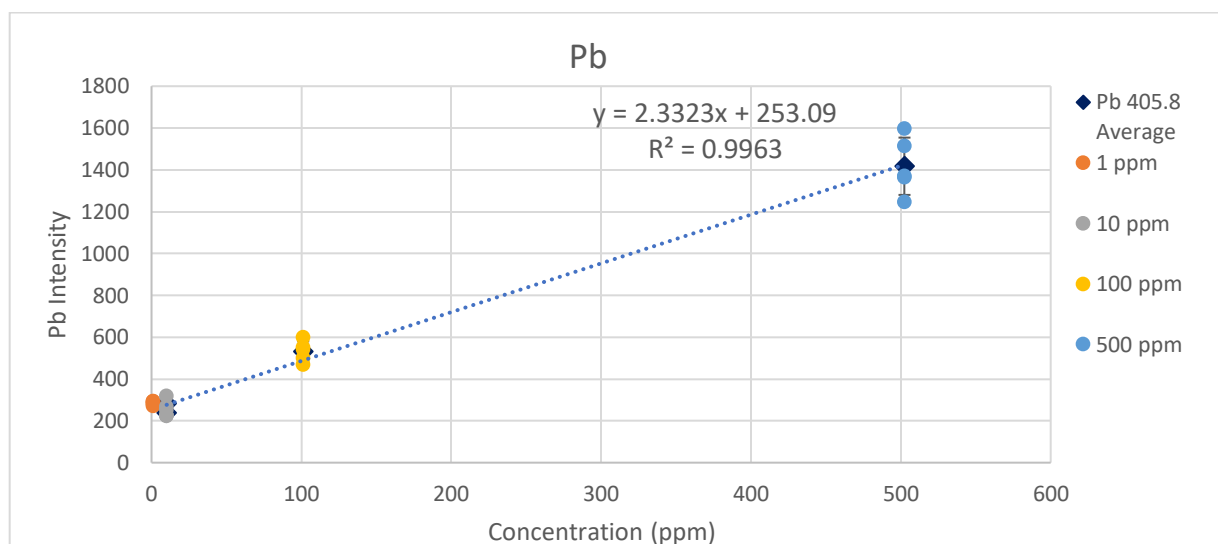
Nickel, Ni

Nickel oxide (NiO) mixtures were prepared in Bi₂O₃ at the target Ni/Bi concentrations listed in Table 2. C-CDE was able to dissolve and analyze the Ni standards. However, the values reported from the destructive analysis were 50% from the target values. Therefore, the standard curve was prepared using the masses and concentrations determined in the preparation of the mixtures and is shown below.



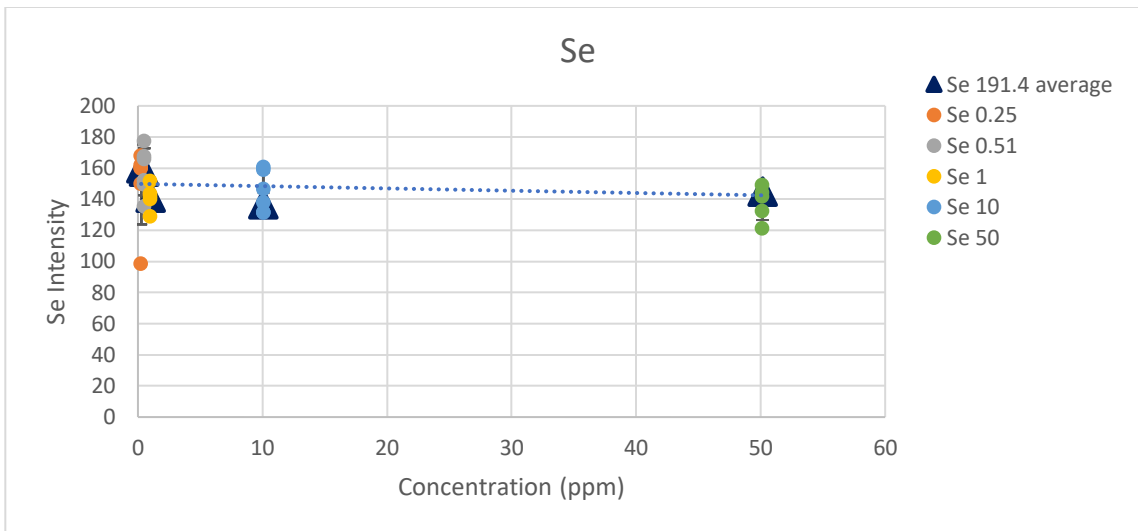
Lead, Pb

Lead oxide (PbO) mixtures were prepared in Bi₂O₃ at the target Pb/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the PbO standards. The standard curve was prepared using the masses and concentrations determined in the preparation of the mixtures and is shown below. The fit using these values is very good, however, preparing more mixtures to add more points between 500 and 100 ppm and 100 and 10 ppm to add to the standard curve could improve the LOD determinations.



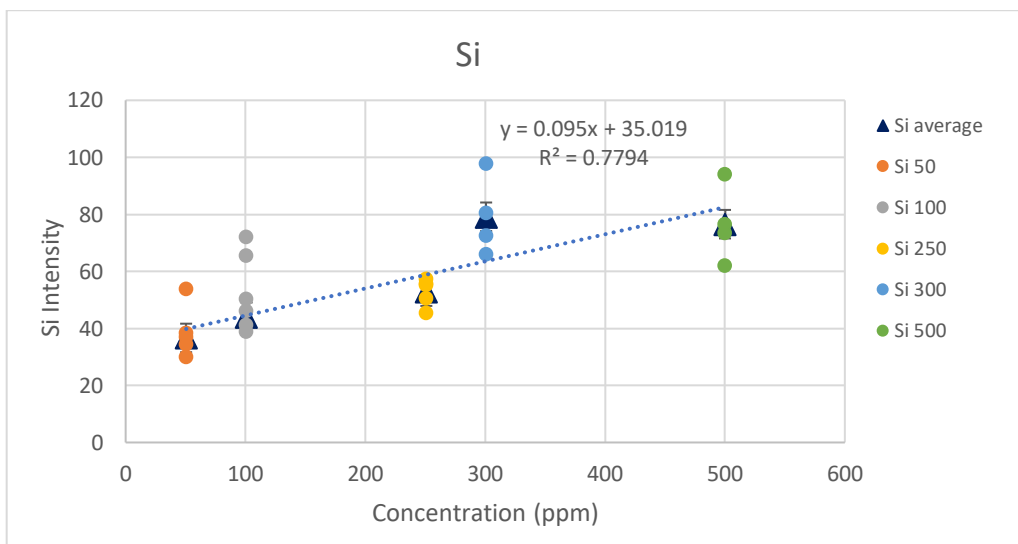
Selenium, Se

Selenium oxide (SeO_2) mixtures were prepared in Bi_2O_3 at the target Se/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the SeO_2 standards. The standard curve was prepared using the masses and concentrations determined in the preparation of the mixtures and is shown below. The standard curve is not too bad, but the intensity values are so low that it was very difficult to determine the LOD. Preparing more mixtures to add more points to the standard curve between 10 and 50 ppm could improve the LOD determinations. It should be noted that Se doesn't have ANY good wavelengths to consider in LIBS measurements that aren't interfered with by other elements, so it is not a good candidate for quantitation by LIBS.



Silicon, Si

Silicon (Si) mixtures were prepared in Bi_2O_3 at the target Si/Bi concentrations listed in Table 2. C-CDE was unable to dissolve and analyze the standards. The standard curve line is very reasonable using the masses and concentrations determined in the preparation of the mixtures. Si has just one wavelength to consider.



Determination of LOD

Several methods were investigated for determining Limits of Detection (LOD) for the analytes of interest. Based on the complexity of the measurements and the uncertainties associated with the preparation of the mixtures, a simple LOD determination was made (Equation 1)³. In equation 1, s is represented by the standard deviation of n measurements at each concentration, and m is the slope of the line determined from measuring the intensity vs concentration. An average LOD was calculated using the two lowest concentration samples.

Eq. 1 $\text{LOD} = 3s/m$

When available, the concentration of the element determined from destructive analysis was used in each standard curve and is represented in the slope used in Eq. 1, unless specified in the above sections. These concentrations are in blue in Table 3. For all other analytes, the concentrations used in the standard curves were determined from the mass data recorded during the preparation of the mixtures. It should also be pointed out that when a standard was prepared that contained two analytes (BaF₂, NaCl, K₂CO₃), the target values and the values used for the standard curves seem to be quite different. However, the target values for only one of the two analytes could be used for the target value calculations. Therefore, in some cases the prepared concentrations for the analyte did not match the target values. In Table 3 those “partner” analytes that were *not* chosen for the measured target calculation are identified with an asterisk.

Table 3. Summary of LOD determinations

Analyte/ Compound	Target Conc (µg/g Bi)	Conc used in LOD (µg/g Bi)	LOD (ppm)
Ag/AgO	0.25	0.26	4.9
	0.50	0.52	
	0.75	0.76	
	10	10.05	
	100	100	
Al/Al ₂ O ₃	15	15.05	64.7
	30	30.09	
	45	45.1	
	60	60.3	
	100	100.23	
As/As ₂ O ₃	1	0.68	4.4
	2.5	2.22	
	5	5.64	
	10	7.78	
	100	112.77	
*Ba/BaF ₂	10	37.33	59.7
	50	93.13	
	100	370.65	
	500	904.58	
	1000	1803.95	

C/Graphite (in CeO ₂)	1	10.65	439.2
	10	104.73	
	100	258.23	
	500	516.23	
	1000	1007.4	
Cd/CdO	0.1	0.04	7.4
	0.5	0.42	
	1	0.78	
	10	7.60	
	100	84.80	
*Cl/NaCl	10	7.79	361.2
	25	38.58	
	100	77.30	
	250	154.22	
	500	231.21	
Cr/Cr ₂ O ₃	5	5.08	22.2
	50	50.44	
	100	101.39	
	500	500.61	
	1000	1003.0	
F/BaF ₂	10	10.33	282.7
	25	25.77	
	100	102.55	
	250	250.28	
	500	499.11	
Fe/Fe ₂ O ₃	10	9.79	16.2
	100	81.48	
	500	421.71	
	1000	831.30	
	2000	1705.47	
Ga/Ga ₂ O ₃	500	385.53	322.6
	1000	778.94	
	10000	10703.95	
	15000	16605.15	
	20000	20382.95	
Hg/HgO	0.05	0.05	46.1
	0.1	0.11	
	0.2	0.21	
	1	1.04	
	100	79.51	
*K/K ₂ CO ₃	1	4.41	39.1
	5	42.00	
	10	777.49	
	25	2779.82	
	50	5025.82	

Mg/MgO	1	3.34	2.1
	5	6.99	
	10	7.75	
	50	42.01	
	100	85.81	
Na/NaCl	5	6.17	95.1
	25	26.27	
	50	35.02	
	100	94.95	
	150	220.41	
Ni/NiO	10	10.22	50.5
	50	51.20	
	100	100.55	
	250	250.93	
	500	500.77	
Pb/PbO	1	1.04	18.9
	5	LIP	
	10	10.19	
	100	101.20	
	500	502.43	
Se/SeO ₂	0.25	0.25	Not measured
	0.5	0.51	
	1	1.02	
	10	10.09	
	50	50.15	
Si/Si	50	50.47	131.0
	100	100.93	
	250	250.86	
	300	300.77	
	500	500.03	

Path Forward

The sections above describe recommendations for improving the standard curves, calibration curve, and LOD determinations for the target analytes in a Bi₂O₃ matrix. An evaluation of the resulting calibration curve was performed with a mixture of nine of the analytes (Ga, Na, Cl, Cr, F, Ba, Mg, Al, and Pb). In all cases the analyte was identified, however, in most cases the measured concentrations are not close to the target values. Since all of the analyte standard curves are included in the overall calibration curve, this result is expected. If more data were collected and the standard curves for each element improved, it is expected that the calibration curve could be used to quantify most of the analytes of interest. There are a few cases where the analytes are not good candidates for LIBS measurements, as described above, and their standard curves would need to be removed from the overall calibration curve. This would also result in an improvement for the quantitative determination of the other analytes. The data also indicates that in some cases the samples weren't homogeneous. In order to improve on the homogeneity, larger samples and

equipment would be needed due to the low target concentrations for several of the elements. In some cases, LIBS might not be a suitable analysis method. A similar study will be performed using a hand held X-Ray Fluorescence (XRF) instrument, which might be better suited for some of these elements.

It is recommended that the operation be moved to PF4 for analysis of the PuO₂ samples. These samples are well characterized by destructive analysis and are also well mixed. It is possible that more than one calibration curve might be needed, based on anticipated concentrations and interferences between elements in the LIBS measurements.

References

1. Kelly Nebgen, Keri Campbell, Sherrod Maxwell, and Beth Judge, C₃Lab, *Bismuth Impurities via ICP-MS and ICP-OES*, September 15, 2020.
2. Abderrahman Atifi, David W. Boyce, John L. DiMeglio, Joel Rosenthal. **Directing the Outcome of CO₂ Reduction at Bismuth Cathodes Using Varied Ionic Liquid Promoters.** *ACS Catalysis*, 2018; 8 (4): 2857 DOI: [10.1021/acscatal.7b03433](https://doi.org/10.1021/acscatal.7b03433)
3. Harris, Daniel C. *Quantitative Chemical Analysis*. 7th ed., W. H. Freeman and Company, 2007.